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IS 6699 (1989): Perfumery Materials - Oil of Cinnamon Leaf
[PCD 18: Natural and Synthetic Fragrance Materials]

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Indian Standard

PERFUMERY MATERIALS — OIL OF CINNAMON LEAF — SPECIFICATION

(First Revision)

भारतीय मानक

सुगन्ध सामग्री – दाल चीनी पत्ती का तेल – विशिष्टि

(पहला पुनरीक्षण)

UDC 665·525·53

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1990 **Price Group 2**

Natural and Synthetic Perfumery Materials Sectional Committee, PCDC 18

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 25 June 1989, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1972. The Committee responsible for the prepration of this standard felt that it should be revised in view of trade practices prevalent in perfumery industry and to suit the material currently being manufactured and sold in the country.

In the present revision, gas chromatographic method for determination of purity of compound which is progressively used in the country, has been included for guidance only under Annex B.

From times immemorial, cinnamon bark derived from *Cinnamomum zeylanicum*, Blume, and cassia derived from *Cinnamon cassia* are known to the perfumery and flavour world. The essential oil derived from bark and leaf of *Cinnamomum zeylanicum*, Blume, are used in perfumery for its spicy and warm woody oriental type odour. The dried bark of cinnamon, both *zeylanicum* and *cassia* varieties are common flavour ingredient and spice used in Indian and international cuisine.

The cinnamon tree is found to be growing commonly in tropical forests. *Cinnamomum zeylancium*, Blume, the true cinnamon is cultivated primarily in Sri Lanka and South India and grows wild in the areas Madagascar and Comores Islands. The cassia varieties are cultivated in China, Vietnam and Indonesia. In addition, in India, a variety of cinnamon *tamala* is also found to be growing primarily in the eastern region. This variety yields *TEJPAT* or *TEJPATTA* which is a spice ingredient mainly used in northern India.

Cinnamon leaf oil of commerce is derived from steam distillation of free and mature leaves of *Cinnamomum zeylanicum*, Blume. The leaves yield the oil which is rich in eugenol content and the bark yields an oil which is rich in cinnammic aldehyde content. Oil of cinnamon leaf is currently produced mainly in India and Sri Lanka, with Sri Lanka accounting for nearly 80 to 90 percent of the total production.

This standard covers cinnamon leaf oil obtained from *Cinnamomum zeylanicum*, Blume only. An International Standard ISO 3524 : 1977 has been published for oil of cinnamon leaf which covers requirements pertaining to oil derived from *Cinnamomum zeylanicum*, Blume, produced in Sri Lanka, Seychelles, South India, Madagascar and Comores Islands.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PERFUMERY MATERIALS — OIL OF CINNAMON LEAF — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for oil of cinnamon leaf obtained from *Cinnamomum zeylanicum*, Blume.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 1980	Relative density (*second revision*)
(Part 5) : 1986	Determination of refractive index
(Part 6) : 1986	Determination of solubility
(Part 11) : 1986	Determination of carbonyl value and content of car bonyl compounds
(Part 12) : 1980	Determination of phenols (*second revision*)
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials
6597 : 1988	Glossary of terms relating to natural and synthetic perfumery materials

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 6597 : 1988 shall apply.

4 REQUIREMENTS

4.1 Description

Oil of cinnamon leaf is a bright yellow liquid having an odour resembling that of clove and cinnamon.

4.1.1 The materials shall be a clear liquid, free from sediment, suspended matter, separated water and adulterants when tested as prescribed in IS 326 (Part 2) : 1980.

4.1.2 The oil shall be tested olfactorily and specially for bye-notes as prescribed in IS 2284 : 1988.

4.2 Solubility

The material shall be soluble in 2 volumes of 70 percent ethanol occasionally with opalescence when tested as prescribed in IS 326 (Part 6) : 1986.

4.3 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in air-tight and preferably amber-coloured glass or aluminium containers, permitting a minimum of air space, or as agreed to between the purchaser and the supplier.

5.1.1 The material shall be protected from light and stored in a cool and dry place.

5.2 Marking

Each container so filled shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and recognized trade-mark, if any;

c) Batch number and date of manufacture; and

d) Net and gross mass of the material.

Table 1 Requirements for Oil of Cinnamon Leaf

(*Clauses* 4.3 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Relative density at 27/27°C	1·030 0 to 1·056 0	IS 326 (Part 3) : 1980
ii)	Refractive index at 27°C	1·525 0 to 1·536 0	IS 326 (Part 5) : 1986
iii)	Phenol (as eugenol)* percent by mass, *Min*	80	IS 326 (Part 12) : 1980
iv)	Aldehyde (as cinnamic aldehyde), percent by mass, *Max* (by hydroxyl ammonium chloride method using 5 g of sample, standing time 15 minutes, Mol Wt 132·2)	4	IS 326 (Part 11) : 1986
v)	Water soluble materials	Nil	Annex A

*Determination of eugenol content by gas chromatographic method has been given for information only under Annex B.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1) : 1984.

7 TESTS

7.1 Tests shall be carried out as prescribed under **4.1**, **4.2** and **4.3**. The appropriate reference to relevant clauses is given in col 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

ANNEX A

[*Table* 1, *Item* (v)]

DETERMINATION OF WATER SOLUBLES IN OIL OF CINNAMON LEAF

A-1 APPARATUS

A-1.1 Graduated Cylinder, 100 ml capacity with ground-glass stopper and graduated in 0·1 ml.

A-2 REAGENTS

A-2.1 Xylene

A-2.2 Sodium Sulphate Solution, 3 percent.

A-3 PROCEDURE

A-3.1 Measure test sample, previously dried with anhydrous sodium sulphate in a clean and dry measuring cylinder so that the volume reads 10 ml, correct to 0·1 ml. Add sufficient xylene to the sample so that the total quantity of the liquid reads 20 ml, correct to 0·1 ml. Pour about 80 ml of sodium sulphate solution warmed to about 40°C. Stopper the cylinder and shake the contents vigorously for about 5 minutes and allow to settle overnight. Read the volume of the top layer of xylene correct to 0·1 ml.

A-3.1.1 The volume of xylene layer shall be not less than 20 ml.

ANNEX B

(*Table* 1, *Foot-note*)

GAS CHROMATOGRAPHIC ANALYSIS FOR OIL OF CINNAMON LEAF

B-0 GENERAL

B-0.1 The chromatographic conditions given here are for guidance only.

B-1 OUTLINE OF THE METHOD

B-1.1 A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constitutents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

B-2 APPARATUS

B-2.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatogram for oil of cinnamon leaf using a chromatograph with the following chromatographic conditions is shown in Fig. 1.

a) *Sample* : Oil of cinnamon leaf

b) *Column*

   1) Material : Copper
   2) Length : 3·65 m
   3) Outer diameter : 0·635 cm
   4) Inner diameter : 0·476 cm
   5) Stationery phase : FFAP*, 10 percent by mass
   6) Solid support : Chromosorb WAW 60-80 mesh

c) *Carrier Gas* : Nitrogen

d) *Conditions*

   1) Column temperature isothermal : 200°C
   2) Injection port temperature : 200°C
   3) Carrier gas flow rate : 20 ml/min
   4) Inlet pressure : 3·5 kg/cm^2

e) *Detector*

   1) Type : Thermal conductivity
   2) Temperature : 285°C

f) *Recorder*

   1) Span : 1 mV
   2) Chart speed : 0·25 cm/min

g) *Attenuation/range* : 32/100

NOTE — This analysis may also be accomplished with columns containing: carbowax 20 M, Diethylene Glycol Succinate (DEGS).

B-3 PROCEDURE

B-3.1 Conduct the flow of the carrier gas and inject the sample (dissolved in suitable solvent) at injection port where it is vapourized and wall mixed with the carrier gas. This is led into the chromatographic column wherein vapourized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding to the amount of particular constituents leaving the column. The detection signals on transmission to the recorder plot the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are derermined.

NOTE — For separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of constituents.

B-4 CALCULATION

B-4.1 Area Measurements (*see* Note)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

---

*Free fatty acid phase (FFAP) is carbowax 20 M treated with nitrophthalic acid.

NOTE — Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine would be of great advantage.

B-4.2 Area Normalization (*see* Note)

By normalizing, it is meant, calculating the percentage composition by measuring the area of each peak and dividing the individual area by total area, for example:

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

Note — Internal standardization may be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

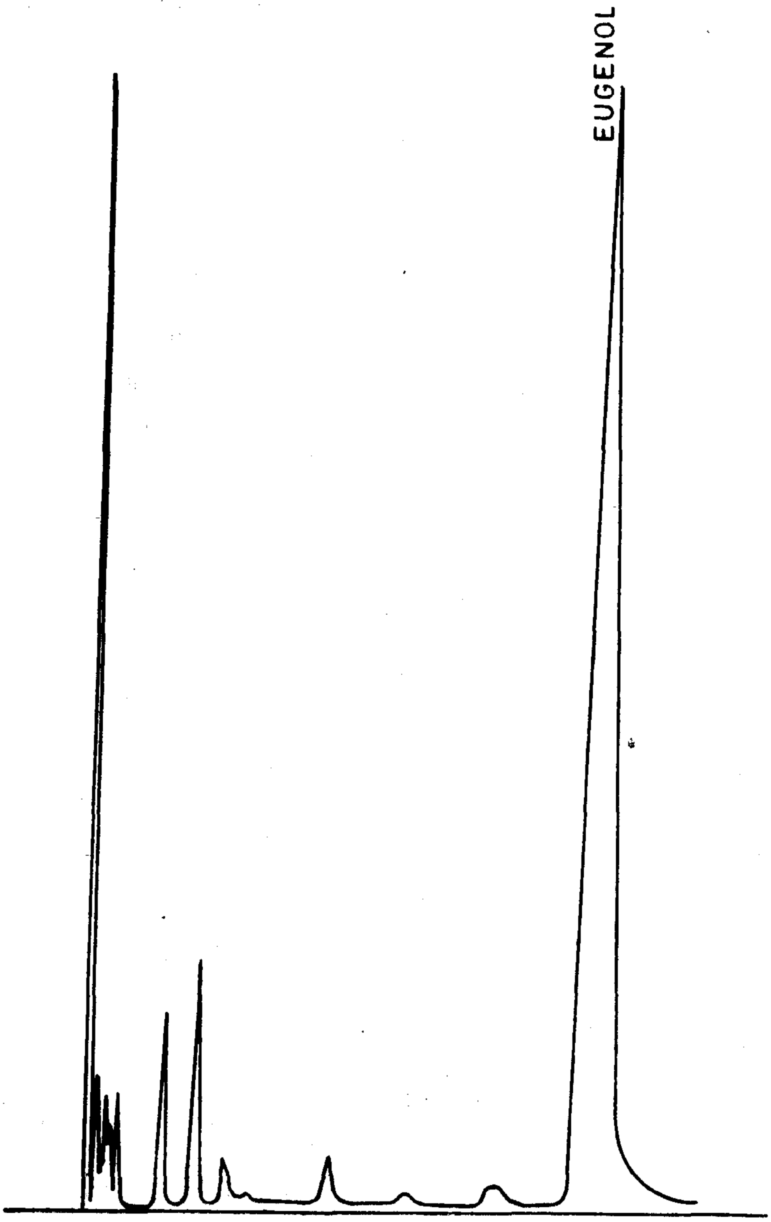

Fig. 1 Typical Chromatogram for Oil of Cinnamon Leaf

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